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(54) BIAXIALLY ORIENTED POLYESTER FILM AND METHOD FOR MANUFACTURING THIS FILM

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a biaxially oriented polyester film which shows upgraded mechanical properties such as the Young's modulus of film and likewise upgraded dimensional stability and especially reliable electromagnetic conversion characteristics, running durability, shelf stability and the like for use as a magnetic recording medium and further, demonstrates the best applications for a heat-sensitive transfer ribbon, a capacitor and the like as well as a method for manufacturing the biaxially oriented polyester film.

SOLUTION: This biaxially oriented polyester film contains a polyester A and a polyetherimido B and has a single glass transition temperature. In addition, this polyester film is characterized in that at least either of the ratio R1M (=IMD/IND) of a Raman peak intensity (IMD) in the longer direction of film to a Raman peak intensity (IND) in the thickness direction of the film or the ratio R1T (=ITD/IND) of the Raman peak intensity (ITD) in the width direction of the film to the Raman peak intensity (IND) in the thickness direction of the film, at 1,776 cm-1 as measured by the laser Raman scattering method, is within the range of 2-20.

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## CLAIMS

[Claim(s)]
[Claim 1]They are polyester (A) and a biaxial orientation polyester film which has a single glass transition temperature containing polyether imide (B), . It can set to 1776-$cm^{-1}$ measured with laser Raman scattering process. The Raman peak intensity to the direction of a film length hand. The Raman peak intensity of ($I_{MD}$) and a film thickness direction. a ratio with ($I_{ND}$) -- $R_{1M}$. ($=I_{MD}/I_{ND}$). or a ratio of the Raman peak intensity ($I_{TD}$) and the Raman peak intensity ($I_{ND}$) of a film thickness direction to a film width direction -- that ranges at least of one side of $R_{1T}$ ($=I_{TD}/I_{ND}$) are 2-20. A biaxial orientation polyester film by which it is characterized.
[Claim 2]The biaxial orientation polyester film according to claim 1 which polyether imide (B) contains one to 50% of the weight.
[Claim 3]The biaxial orientation polyester film according to claim 1 or 2 whose sums of Young's modulus of a longitudinal direction and the cross direction are 10-25 (GPa).
[Claim 4]The biaxial orientation polyester film according to any one of claims 1 to 3 each of 100 ** of a longitudinal direction and the cross direction and whose heat shrinkage rates [ / in 30 minutes ] is 0.01 to 2.0%.
[Claim 5]The biaxial orientation polyester film according to any one of claims 1 to 4 which is that to which polyester (A) uses an ethylene terephthalate unit as a main ingredient.
[Claim 6]They are polyester (A) and a biaxial orientation polyester film which has a single glass transition temperature containing polyether imide (B), . It can set to 1615-$cm^{-1}$ measured with laser Raman scattering process. The Raman peak intensity to the direction of a film length hand. The Raman peak intensity of ($I_{2MD}$) and a film thickness direction. a ratio with ($I_{2ND}$) -- $R_{2M}$. ($=I_{2MD}/I_{2ND}$). Or the Raman peak intensity to a film width direction. a ratio of ($I_{2TD}$) and the Raman peak intensity ($I_{2ND}$) of a film thickness direction -- the biaxial orientation polyester film according to claim 5 ranges at least of whose one side of $R_{2T}$ ($=I_{2TD}/I_{2ND}$) are 5-45.
[Claim 7]The biaxial orientation polyester film according to claim 5 or 6 whose extrapolation glass transition starting temperature (Tg-onset) is 90-150 **.
[Claim 8]In a manufacturing method of a biaxial orientation polyester film which comes to carry out biaxial orientation after carrying out cooling solidification of the melting polymer obtained by melting extrusion and

fabricating to a sheet shaped, Polyester (A) and melting polymer containing polyether imide (B) are dissolved by melting extrusion, Carry out cooling solidification, fabricate to a sheet shaped, and this sheet-shaped molding to a longitudinal direction Three to 10 times, A manufacturing method of a biaxial orientation polyester film which extends by one 3 to 10 times the magnification of this crosswise, and is characterized by a thing of a longitudinal direction or the cross direction extended 1.1 to twice to one way at least at temperature of 180 ** - 250 ** after an appropriate time.

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## DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the biaxial orientation polyester film which raised substantially the physical properties and quality of conventional polyester film, and its manufacturing method.
[0002]It is related with the biaxial orientation polyester film which was excellent in rigidity, tough nature, dimensional stability, etc., for example, was specifically dramatically suitable as various kinds of films for industrial materials the object for magnetic recording media, the object for capacitors, for thermal transfer ribbons, etc.
[0003]
[Description of the Prior Art]Which feature [ a mass production of the film of a large area which is not obtained from other raw materials is possible for polyester film, and ] that can give the intensity, endurance, transparency, pliability, and a surface characteristic is harnessed, It is used in the various fields which have demand in the large quantities the various objects for industrial materials the object for magnetic recording media, the object for capacitors, the object for thermal transfer ribbons, for [ for thermal mimeograph printing ] stencil paper, etc., the object for agriculture, the object for a package, for building materials, etc.
[0004]Also in it, the biaxial orientation polyester film is used in various fields from a viewpoint of a mechanical characteristic or dimensional stability, etc., and is especially useful as a base film for magnetic recording media. In the object for magnetic recording media, much more thin film-ization of the base film is demanded especially in recent years for the weight saving of equipments, a miniaturization, and record [ prolonged ]-izing. Also in the object for thermal transfer ribbons, the object for capacitors, or the object for thermal mimeograph printing stencil paper, the tendency of thin-film-izing is dramatically strong in recent years.
[0005]However, if a film is thin-film-ized, a mechanical strength becomes insufficient and the nerve of a film becomes weak, or since it becomes easy to be extended, by the object for magnetic recording media, it becomes that it is easy to receive a tape damage, for example, or a head touch will get worse and a magnetic parametric performance will fall. When a film is thin-film-ized, at the object for thermal transfer ribbons, the surface smoothness of the ribbon at the time of printing is not maintained, printing nonuniformity and fault transfer occur, and there is a problem that dielectric breakdown voltage falls, by the object for capacitors.

[0006]In such thin film-ized-oriented, improvement in mechanical characteristics, such as tractive characteristics which are represented by Young's modulus, is desired.
[0007]Therefore, high intensity-ization of a film has been considered by various methods from the former. As the technique of high-intensity-izing of a biaxially oriented polyester film in which it is generally known, For example, what is called the re-length extending method that extends again the film extended for length and horizontal two way types to a lengthwise direction, and is high-intensity-ized to a lengthwise direction is common (for example, JP,42-9270,B, JP,43-3040,B, JP,46-1119,B, JP,46-1120,B, etc.).
[0008]After performing above-mentioned re-length extension, the re-**** lateral orientation method for extending in a transverse direction again is proposed to also give intensity to a transverse direction (for example, JP,50-133276,A, JP,55-22915,A, etc.). Further extension is performed to the lengthwise direction of a film in two or more steps, and the vertical multi stage extending method which extends in the transverse direction of a film is proposed succeedingly (for example, JP,52-33666,B, JP,57-49377,B, etc.).
[0009]However, the high intensity-ized polyester film obtained by such conventional technology, For example, since a dimensional change is carried out, a gap arises in a recording track and an error occurs by stress elongation deformation or an environmental condition in the object for magnetic recording media at the time of record reproduction, The actual condition is that there are problems -- a desired magnetic parametric performance is not acquired -- and the technical problem is left behind on the occasion of application to a mass high-density magnetic recording tape.
[0010]On the other hand, also in the past about polyester and the constituent of polyether imide (PEI), there was description, for example, as polyester, If the constituent of the various mixture ratio is created using polyethylene terephthalate (PET), . It is shown that glass transition temperature rises with the increase in the weight fraction of PEI. for example, "JOURNAL of APPLIEDPOLYMER SCIENCE" -- 935 - 937 pages 48 volumes in 1993, "Macromolecules" 1995, 28 volumes, 2845 - 2851 pages, "POLYMER"1997 year, 38 volumes, 4043 - 4048 pages", etc. However, a report of the biaxial oriented film which consists of a mixture of PET and PEI is not made, furthermore it is not known at all about the mechanical characteristic or dimensional stability of this biaxial oriented film, and the actual condition is not inquired.
[0011]
[Problem(s) to be Solved by the Invention]When it is used the purpose of this invention being to provide the quality biaxial orientation polyester film excellent in mechanical strength and dimensional stability, such as Young's modulus, and its manufacturing method, and having carried out especially the base film for magnetic recording media, It excels in a magnetic parametric performance, running durability, or preservation stability, is suitable for the base film for high-density magnetic recording tapes, and is providing the still more suitable biaxial orientation polyester film as the object for thermal-ink-transfer-printing ribbons, and an object for capacitors, and its manufacturing method.
[0012]
[Means for Solving the Problem]A biaxial orientation polyester film of this invention in alignment with the above-mentioned purpose, They are polyester (A) and a biaxial orientation polyester film which has a single glass transition temperature containing polyether imide (B), . It can set to 1776-$cm^{-1}$ measured with laser Raman scattering process. The Raman peak intensity to the direction of a film length hand. The

Raman peak intensity of ($I_{MD}$) and a film thickness direction. a ratio with ($I_{ND}$) -- $R_{1M}$. ($=I_{MD}/I_{ND}$). or a ratio of the Raman peak intensity ($I_{TD}$) and the Raman peak intensity ($I_{ND}$) of a film thickness direction to a film width direction -- it is characterized by ranges at least of one side of $R_{1T}$ ($=I_{TD}/I_{ND}$) being 2-20.

[0013]A manufacturing method of a biaxially oriented polyester film of this invention, In a manufacturing method of a biaxial orientation polyester film which comes to carry out biaxial orientation after carrying out cooling solidification of the melting polymer obtained by melting extrusion and fabricating to a sheet shaped, Polyester (A) and melting polymer containing polyether imide (B) are dissolved by melting extrusion, Cooling solidification is carried out and it fabricates to a sheet shaped, and it extends by three to 10 times to a longitudinal direction, this sheet-shaped molding is extended crosswise by one 3 to 10 times the magnification of this, and it is characterized by a thing of a longitudinal direction or the cross direction extended 1.1 to twice to one way at least at temperature of 180 ** - 250 ** after an appropriate time.

[0014]

[Embodiment of the Invention]The polyester (A) which constitutes the biaxial orientation polyester film of this invention comprises acid components and diol components, such as aromatic dicarboxylic acid, alicycle fellows dicarboxylic acid, or aliphatic dicarboxylic acid, for example.

[0015]As an aromatic dicarboxylic acid component, for example Terephthalic acid, isophthalic acid, Phthalic acid, 1, 4 **NAFUTA range carboxylic acid, 1, 5 **NAFUTA range carboxylic acid, 2, 6 **NAFUTA range carboxylic acid, 4, and 4' **JIFE nil dicarboxylic acid, **JIFE nil ether dicarboxylic acid, and 4 and 4 '4, 4' **JIFE nil sulfone dicarboxylic acid etc. can be used, and terephthalic acid, phthalic acid, 2, and 6 **NAFUTA range carboxylic acid can be used preferably especially. As an alicycle fellows dicarboxylic acid component, cyclohexanedicarboxylic acid etc. can be used, for example. As an aliphatic dicarboxylic acid component, adipic acid, suberic acid, sebacic acid, dodecane dione acid, etc. can be used, for example. These acid components may use only a kind and may use two or more sorts together.

[0016]As a diol component, for example Ethylene glycol, 1, 2-propanediol, 1,3-propanediol, neopentyl glycol, 1,3-butanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 1, 2 **SHIKURO hexanedimethanol, 1, 3 **SHIKURO hexanedimethanol, 1, 4 **SHIKURO hexanedimethanol, a diethylene glycol, triethylene glycol, Can use polyalkylene glycol, 2,2' **BISU (4'-beta **HIDOROKISHI ethoxyphenyl) propane, etc., and especially preferably, Ethylene glycol, 1,4-butanediol, 1, 4 **SHIKURO hexanedimethanol, a diethylene glycol, etc. can be used, and ethylene glycol etc. can be used especially preferably. These diol components may use only a kind and may use two or more sorts together.

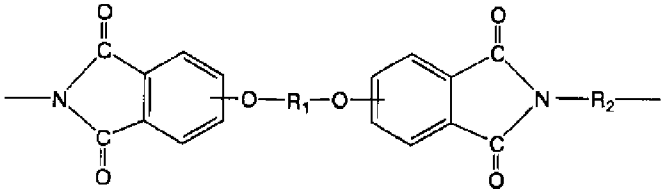
[0017]Copolymerization of the monofunctional compounds, such as lauryl alcohol and a phenyl isocyanate, may be carried out to polyester, and, 3 organic-functions compounds, such as trimellitic acid, pyromellitic acid, glycerol, pentaerythritol, 2, and 4 **JIOKISHI benzoic acid, etc. carry out neither branching nor bridge construction too much, but copolymerization of the polymer may be carried out by within the limits which is a line substantially. Furthermore, in addition to an acid component and a diol component, para-hydroxybenzoic acid, m-hydroxybenzoic acid, If it is a small quantity which is a grade by which the effect of this invention is not spoiled, copolymerization of aromatic hydroxycarboxylic acid, such as 2,6-hydroxynaphthoic acid, and p-aminophenol, the p-aminobenzoic acid, etc. will be carried out further.

[0018]Although polyester (A) in particular of this invention is not limited, from points, such as mechanical strength, productivity, and handling nature. The thing which was chosen from the group which consists of

polyester which makes a main constituent ethylene terephthalate and/or the ethylene 2, and 6 **NAFUTA range carboxylate unit, and those denaturation objects and which is a kind at least is preferred. Also among these, especially the polyester that makes an ethylene terephthalate unit a main constituent (it contains especially 80% of the weight or more) is desirable. The polyester which makes an ethylene terephthalate unit a main constituent is because it is easy to carry out extrusion-molding processing and there are few film tears at the time of film production than the polyester which makes an ethylene-2,6-naphthalene dicarboxylate unit a main constituent. However, there is an advantage that compatibility with polyether imide is good in the latter.

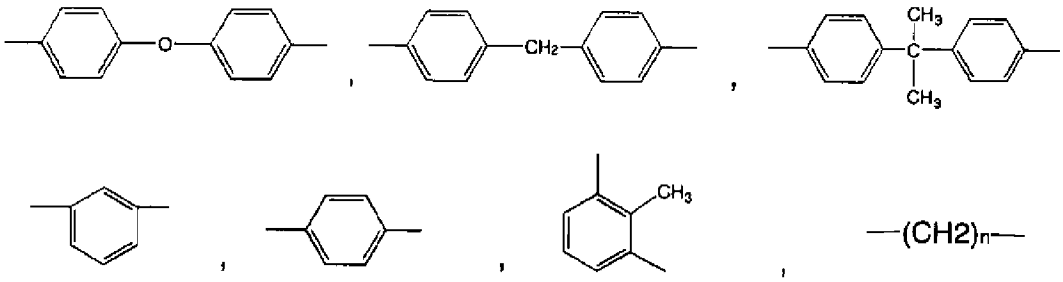
[0019]Although the polyether imide (B) in particular of this invention is not limited, it is preferred that it is a structural unit which contains an ether bond in a polyimide constituent from points, such as melt molding nature with polyester (A) and handling nature, for example as shown in a following general formula.

[Formula 1]



However, divalent aromatic residue in which divalent aromatic series or aliphatic series residue;$R_2$ in which the above-mentioned $R_1$ in a formula has 6-30 carbon atoms has 6-30 carbon atoms, It is the divalent organic group chosen from a group which consists of a polydyorganosiloxane group by which chain termination was carried out by alkylene group which has 2-20 carbon atoms, cyclo alkylene group which has 2-20 carbon atoms, and an alkylene group which has 2-8 carbon atoms.

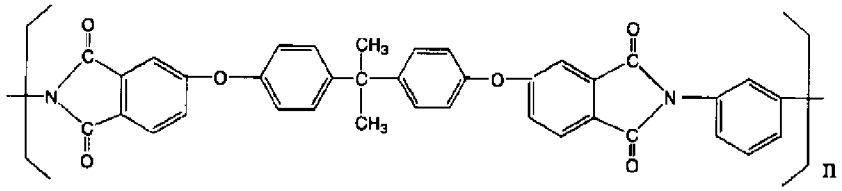
[0020]Aromatic residue shown in a following formula group as the above-mentioned $R_1$ and $R_2$, for example [Formula 2]



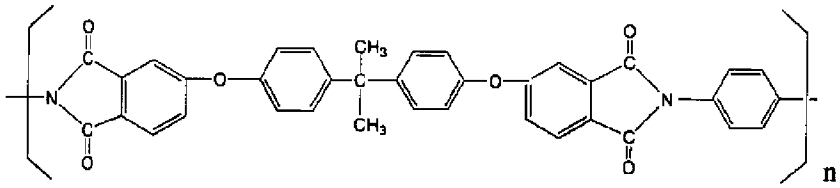
******** -- things are made.

[0021]In this invention, from viewpoints of compatibility with polyester (A), cost, melt molding nature, etc. A condensate with 2,2-screw [4-(2,3-dicarboxyphenoxy) phenyl] propane dianhydride which has a structural unit shown with a following formula, m-phenylenediamine, or p-phenylene diamine is preferred.

[Formula 3]



または



This polyether imide is a brand name of "Ultem" (registered trademark), and it is more nearly available than a GE plastics company in it.

[0022]Compatibility here means that the glass transition temperature (Tg) of the obtained chip is single. Thus, it is generally known that Tg when both dissolve exists between Tg of polyester (A) and Tg of the pellet (B) of polyether imide. According to JIS K7121, it can ask for the glass transition temperature as used in the field of this invention from the thermal flux gap at the time of the temperature up in a differential scanning calorimetry. When it is hard to judge only by the method by a differential scanning calorimetry, morphological methods, such as dynamic viscoelasticity measurement or microscope observation, may be used together. When judging glass transition temperature by a differential scanning calorimetry, it is also effective to use the temperature becoming [ irregular ] method and a high sensitivity method. When this film has two or more glass transition temperature, polyester (A) and polyether imide (B) do not dissolve in a film, and the effect of this invention is not acquired. The biaxial orientation polyester film which has a single glass transition point temperature by this invention has pointed out having a special feature which requires at least one layer of a film, when it is a laminated film more than two-layer. therefore, the film from which a glass transition point differs may be laminated in the range which does not bar an effect of the invention on the film of this invention. However, since film manufacture will become difficult if the glass transition points between each class laminated differ not much, as for the difference of a glass transition point, 50 ** or less is preferred, and it is more preferred. [ of 30 ** or less ] However, a coating layer does not have that it is not this limitation until it says it.

[0023]In this invention, although the stage in particular to add polyether imide (B) in polyester (A) is not limited, it may add before a polymerization of polyester, for example, an esterification reaction, and it may be added before melting extrusion after a polymerization. Polyester (A) and polyether imide (B) may be pelletized before melting extrusion.

[0024]In a biaxial orientation polyester film of this invention. . It can set to 1776-$cm^{-1}$ measured with laser Raman scattering process. The Raman peak intensity to the direction of a film length hand. The Raman peak intensity of ($I_{MD}$) and a film thickness direction. a ratio with ($I_{ND}$) -- $R_{1M}$. a ratio of the Raman peak intensity ($I_{TD}$) and the Raman peak intensity ($I_{ND}$) of a film thickness direction to ($=I_{MD}/I_{ND}$) or a film width

direction -- ranges at least of one side of $R_{1T}$ ($=I_{TD}/I_{ND}$) are 2-20. The Raman peak intensity in 1776-$cm^{-1}$ measured with laser Raman scattering process in this invention, Strength of orientation of polyether imide (B) to the measurement direction is reflected, and intensity ratio $R_{1M}$ or $R_{1T}$ serves as an index of strength of relative orientation within a film plane. It can ask for this by measuring Raman scattering light at the time of hitting a laser beam to a film. In the Raman spectrum, a Raman band of 1776-$cm^{-1}$ belongs to C=O stretching vibration of a carbonyl group under imide bonding. The desirable ranges of Raman peak intensity ratio $R_{1M}$ or $R_{1T}$ are 2.5-10, and ranges of a still more desirable range are 3-8. Since orientation within a film plane of polyether imide (B) is not enough in this Raman peak intensity ratio $R_{1M}$ or $R_{1T}$ being less than two and intensity of a film is not enough, When the purpose of this invention cannot be attained, for example, it uses for a magnetic-recording-medium tape etc., it may be inferior to a magnetic parametric performance or running durability. On the other hand, if this Raman peak intensity ratio $R_{1M}$ or $R_{1T}$ exceeds 20, it is inferior to the dimensional stability of a film, or tear propagation resistance-proof will be small and it will become easy to produce a film tear.

[0025]In a biaxial orientation polyester film of this invention. Although not limited in particular, a ratio with the Raman peak intensity ($I_{45}$) to the Raman peak intensity ($I_{MD}$) and a longitudinal direction to the direction of a film length hand in 1776-$cm^{-1}$ measured with laser Raman scattering process, and a direction which makes 45 degrees -- $R_{M45}$. ($=I_{MD}/I_{45}$). or a ratio with the Raman peak intensity ($I_{45}$) to the Raman peak intensity ($I_{TD}$) and the cross direction to a film width direction, and a direction which makes 45 degrees -- it is preferred that ranges of each $R_{T45}$ ($=I_{TD}/I_{45}$) are 1.5-5. This Raman peak intensity ratio $R_{M45}$ and $R_{T45}$ have the more preferred range of 1.8-4, and their range of 2-3 is still more preferred. This Raman peak intensity ratio $R_{M45}$ and $R_{T45}$ that it is less than 1.5 Since intensity of a longitudinal direction of a film or the cross direction is not enough, When the purpose of this invention cannot be attained, for example, it uses for a magnetic-recording-medium tape etc., it may be inferior to a magnetic parametric performance or running durability. On the other hand, if this Raman peak intensity ratio $R_{M45}$ and $R_{T45}$ exceed five, it is inferior to the dimensional stability of a film, or tear propagation resistance-proof will be small and it will become easy to produce a film tear.

[0026]In a biaxial orientation polyester film of this invention. Although not limited in particular, It is preferred that it is a range whose half peak width of a circumferencial direction of a diffraction peak of a crystal face of this biaxial orientation polyester main chain direction acquired when the normal is rotated for this biaxial orientation polyester film as an axis in crystal orientation analysis by the diffractometer method of a wide angle X diffraction is 30 to 80 degrees. Half breadth of a circumferencial direction of a diffraction peak of a crystal face of the direction of a polyester main chain is a thing showing breadth of distribution of the direction of orientation of a crystal of a biaxial orientation polyester film, When this half peak width is less than 30 degrees, it is inferior to the dimensional stability of a film, and preservation stability gets worse or it becomes [ tear propagation resistance of a film becomes small and ] easy to produce a tape fracture. When half breadth exceeds 80 degrees, intensity of a film is not enough and may be unable to attain the

purpose of this invention. Here with a crystal face of the direction of a polyester main chain. In a crystal face detected as a diffraction peak by a wide angle X-ray diffractometer method, the normal is a crystal face nearest to the direction of a polyester main chain, for example, it is a field in polyethylene terephthalate at a field, the polyethylene 2, and 6-naphthalate (-306). (-105) If it is an object for data storage, for example when using for magnetic recording media, its range of 30 to 55 degrees is more preferred, and if said half peak width is an object for the videotapes of helical scan systems, such as a digital video, its range of 55 to 80 degrees is more preferred.

[0027]Although a crystal size in particular of the direction of a polyester main chain acquired from a wide angle X-ray diffraction method about a biaxial oriented film of this invention is not limited, it is preferred that it is the range of 90 to [ more than ] 40 A or less. Here, the direction of a polyester main chain is a normal line direction of a crystal face nearest to the direction of a polyester main chain, for example, is a normal line direction of a field in polyethylene terephthalate at a field, the polyethylene 2, and 6-naphthalate (-306). (-105) In less than 40 A, elongation deformation of a tape becomes large, and this crystal size also tends to generate an edge damage, and preservation stability after tape processing gets worse. When a crystal size exceeds 90 A, occurrence frequency of a tape fracture may become high.Although it changes with polyester to be used, when it is polyethylene terephthalate, this crystal size has the more preferred range of 85 to [ more than ] 45 A or less, and its range of 80 to [ more than ] 50 A or less is still more preferred. When polyester to be used is the polyethylene 2 and 6-naphthalate, the range of 65 to [ more than ] 35 A or less is still more preferred.

[0028]Although content in particular of polyether imide (B) of a biaxial orientation polyester film of this invention is not limited, it is preferred that it is in 1 to 50% of the weight of a range. Still more preferably, it is 5 to 30% of the weight of a range, and is 10 to 25% of the weight of a range more preferably. Since melt viscosity of polyester (A) and polyether imide (B) differs greatly, if content of polyether imide (B) is less than 1 % of the weight, it is sometimes difficult to obtain kneading sufficient with an extrusion machine and to dissolve mutually. If content of polyether imide (B) is the quantity exceeding 50 % of the weight, in order that extrusion-molding processing may reveal sufficient intensity for difficult and further obtained polyester film, it is sometimes difficult to perform a stretching process.

[0029]Although the sum in particular of Young's modulus of a longitudinal direction of a biaxial orientation polyester film of this invention and crosswise Young's modulus is not limited, it is preferred that it is the range of 10 - 25GPa, and it is 13 - 20GPa still more preferably 12 to 22 GPa more preferably. For tension which will be received from a magnetic-recording head and a guide pin at the time of a run, for example when using for magnetic recording media if the sum of this Young's modulus is less than 10 GPa, It becomes easy to produce elongation deformation in magnetic tape, it may have an adverse effect on a magnetic parametric performance (output characteristics) further, and use may not be borne practically. A film on which the sum of this Young's modulus exceeds 25GPa may be industrially difficult to manufacture, or tearing resistance and dimensional stability of a film may fall remarkably.

[0030]Although temperature of 100 ** of a longitudinal direction of a biaxial orientation polyester film of this invention and the cross direction and a heat shrinkage rate [ / in 30 minutes ] in particular are not limited, it is preferred from a viewpoint of the elongation deformation nature of a tape, and preservability that each is 0.01 to 2.0%. More preferably, it is 0.01 to 1.5% and is 0.01 to 1.0% still more preferably. When a heat shrinkage rate with a temperature of 100 ** exceeds 2.0%, In [ dimensional stability may become is easy to

be spoiled, for example, ] an object for magnetic recording media, Heat modification of a tape may become being easy to happen at the time of temperature up of magnetic tape by frictional heat of a heat history in a film processing process of applying a magnetic layer of a base film, magnetic tape at the time of a run, and a magnetic-recording head, or the preservability of a tape may get worse. When a heat shrinkage rate with a temperature of 100 ** is less than 0.01%, a film may expand and wrinkles may occur.

[0031]Although a biaxial orientation polyester film in particular of this invention is not limited, when an ingredient which consists of polyester (A) which uses an ethylene terephthalate unit as a main ingredient, and polyether imide (B) is contained, To 1615-$cm^{-1}$ measured with laser Raman scattering process. The Raman peak intensity to the direction of a film length hand which can be set. The Raman peak intensity of ($I_{2MD}$) and a film thickness direction. ($I_{2ND}$) and a ratio -- $R_{2M}$. ($=I_{2MD}/I_{2ND}$). or a ratio of the Raman peak intensity ($I_{2TD}$) and the Raman peak intensity ($I_{2ND}$) of a film thickness direction to a film width direction -- ranges at least of one side of $R_{2T}$ ($=I_{2TD}/I_{2ND}$) are 5-45. The Raman peak intensity in 1615 $cm^{-1}$ measured with laser Raman scattering process in this invention, It is an index which shows strength of orientation of polyester (A) to the measurement direction, and intensity ratio $R_{2M}$ or $R_{2T}$ serves as an index of strength of orientation which receives in a film plane in parallel. In the Raman spectrum, a Raman band of 1615 $cm^{-1}$ belongs to C=C stretching vibration of the benzene ring. The desirable ranges of Raman peak intensity ratio $R_{2M}$ or $R_{2T}$ are 6-40, and ranges of a still more desirable range are 7-30. Since orientation to a film parallel direction of polyester (A) is not enough in this Raman peak intensity ratio $R_{2M}$ or $R_{2T}$ being less than five and intensity of a film is not enough, When the purpose of this invention cannot be attained, for example, it uses for a magnetic-recording-medium tape etc., it may be inferior to a magnetic parametric performance or running durability. On the other hand, if this Raman peak intensity ratio $R_{2M}$ or $R_{2T}$ exceeds 45, it is inferior to the dimensional stability of a film, or tear propagation resistance-proof will be small and it will become easy to produce a film tear.

[0032]Although a biaxial orientation polyester film in particular of this invention is not limited, when an ingredient which consists of polyester (A) which uses an ethylene terephthalate unit as a main ingredient, and polyether imide (B) is contained, It is preferred that the extrapolation glass transition starting temperature (Tg-onset) is 90-150 **. Tg-onset is that there is 95-130 ** within the limits of 100-120 ** still more preferably more preferably. If Tg-onset is less than 90 **, an effect of this invention is sometimes small about improvement in dimensional stability of a film. If Tg-onset is the temperature over 150 **, it may be inferior in respect of fabricating operations, such as melt molding nature and orientation processability.

[0033]A monolayer or a laminated structure more than two-layer may be sufficient as a biaxial orientation polyester film of this invention. Although not limited in particular, it is more desirable to be a laminated structure more than two-layer. If particles are made to contain when using, for example that it is a monolayer as an object for magnetic recording media, a surface projection may not gather but a magnetic parametric performance and performance traverse may get worse. In the case of three layers, an effect of this invention becomes much more good, and it is desirable. Although thickness in particular of the outermost layer is not limited, an effect of this invention becomes much more good, and it is preferred that it is 0.1 to 10 times the pitch diameter of particles contained in the outermost layer. It is because there is a

possibility of becoming poor [ a magnetic parametric performance ] if less than a lower limit of this range, and there is poor fear of performance traverse on the other hand when upper limit of this range is exceeded. When making it laminate, at least one layer consists of polyester (A) and polyether imides (B) in a laminated structure more than two-layer. Although other layers in particular are not limited, it is preferably illustrated by polyester and as the polyester, Although not limited in particular, it is especially desirable when using as a major constituent at least a kind of structural unit chosen from ethylene terephthalate, the ethylene- alpha, beta-bis(2-KURORU phenoxy)ethane-4,4'-dicarboxylate, the ethylene 2, and 6-naphthalate unit.

[0034]When a layer which consists of polyester (A) and polyether imide (B) is allotted to a inner layer, effects, such as preservability and improvement in tensile strength, are acquired. In that case, as for the thickness, it is preferred that it is not less than 80% of the whole thickness. When it allots an outer layer, improvement in running durability has an effect. In that case, as for the thickness, it is preferred that it is 0.1 micrometers or more.

[0035]Although intrinsic viscosity in particular of polyester (A) of this invention is not limited, it is 0.60-2.0 (dl/g) from a viewpoint of the stability of a film fabricating operation, or mixing nature with polyether imide (B) that it is the range of 0.55-3.0 (dl/g) desirable still more preferably. Although intrinsic viscosity in particular of a biaxial orientation polyester film is not limited, it is desirable still more preferred from viewpoints of the stability of a film fabricating operation, dimensional stability, etc. that it is the range of 0.50-2.0 (dl/g), and it is 0.55-1.0 (dl/g).

[0036]Organic lubricant, such as a thermostabilizer, an antioxidant, an ultraviolet ray absorbent, a spray for preventing static electricity, fire retardant, paints, a color, fatty acid ester, and a wax, etc. may be added within limits from which a biaxial orientation polyester film of this invention does not prevent this invention. It is [ in / an object for magnetic recording media, etc. / for example, ] useful, if an inorganic particle, organic particles, etc. are added to the outermost layer of a laminated film in order to give smoothability, abrasion resistance, scratch-proof nature, etc. to a film surface. As this additive, clay, mica, titanium oxide, calcium carbonate, Carrion, talc, a wet type or dry process silica, colloidal silica, calcium phosphate, There are what is called internal particles that deposit according to organic particles which make a constituent inorganic particles, such as barium sulfate, alumina, and zirconia, acrylic acid, styrene, etc., a catalyst added to polyester polymerization reaction time, etc., a surface-active agent, etc.

[0037]Although a use in particular of a biaxial orientation polyester film of this invention is not limited, it is used for an object for magnetic recording media, an object for capacitors, an object for thermal-ink-transfer-printing ribbons, and thermal mimeograph printing stencil paper.

[0038]Although thickness in particular of a biaxial orientation polyester film of this invention is not limited, a range of 1000 micrometers or less is 0.5-500 micrometers desirable still more preferably. Although it can determine suitably according to a use and the purpose like the after-mentioned, the range of 0.5-20 micrometers is preferred, for example. By an object for magnetic recording media, it is suitable for a tape for high-density magnetic recording, for example, a base film for data storage, especially, and not less than 70 GB is not less than 100 GB still more preferably more preferably more than 30 GB (G byte) as this data recording capacity. As linear storage density, 34 K bytes/cm or more of 25 K bytes/cm or more are 39 K bytes/cm or more further more preferably more preferably. In an object for magnetic recording materials, the range of film thickness of 3-9 micrometers is usually preferred at 1-15 micrometers, an object for data,

or an object for digital videos for coating type magnetic recording media in 2-10 micrometers, an object for data, or an object for digital videos for vapor-deposited type magnetic recording media. A 0.5-15-micrometer film is preferably applied to capacitors, and it becomes the thing excellent in stability of dielectric breakdown voltage and dielectric characteristics. without a 1-6-micrometer film is preferably applied to a thermal transfer ribbon use, there are no wrinkles at the time of printing in it and it produces fault transfer of printing unevenness or ink -- a paragraph -- minute printing can carry out.

[0039]A biaxial orientation polyester film of this invention may laminate other polymer layers, for example, polyolefine, polyamide, polyvinylidene chlorides, and acrylic polymer via layers, such as direct or adhesives, to this.

[0040]A biaxial orientation polyester film of this invention may perform arbitrary processings of heat treatment, shaping, a surface treatment, a lamination, coating, printing, embossing, etching, etc. if needed.

[0041]A manufacturing method of a biaxially oriented polyester film of this invention, In a manufacturing method of a biaxial orientation polyester film which comes to carry out biaxial orientation after carrying out cooling solidification of the melting polymer obtained by melting extrusion and fabricating to a sheet shaped, Polyester (A) and melting polymer containing polyether imide (B) are dissolved by melting extrusion, Cooling solidification is carried out and it fabricates to a sheet shaped, and it extends by three to 10 times to a longitudinal direction, this sheet-shaped molding is extended crosswise by one 3 to 10 times the magnification of this (first half extension), and it is a thing (second-half extension) of a longitudinal direction or the cross direction extended 1.1 to twice to one way at least at temperature of 180 ** - 250 ** after an appropriate time. First half extension of more desirable extension conditions is 3.5 to 9 times the magnification of this to 3.5 to 9 times, and the cross direction at a longitudinal direction, and still more desirable conditions are 4 to 8 times the magnifications of this to four to 8 times, and the cross direction at a longitudinal direction. After extending a longitudinal direction and crosswise, as second-half extension, Preferably, it is a 200-230 ** temperature requirement still more preferably, and 190-240 ** of 180-250 ** is a thing of a longitudinal direction or the cross direction preferably extended 1.2 to 1.5 times still more preferably 1.15 to 1.8 times 1.1 to twice to one way at least. This extension is indispensable in a method of this invention. However, it may have processes, such as heat treatment and relaxation, in the middle of first half extension and second-half extension if needed. When dissolving polyester (A) and polyether imide (B), although the stage in particular to add polyether imide (B) in polyester (A) is not limited, it may add before a polymerization of polyester, for example, an esterification reaction, and it may be added before melting extrusion after a polymerization. Especially, it is preferred from a viewpoint of melt molding nature to pelletize polyester (A) and polyether imide (B) and to make it master chips in front of melting extrusion. It is especially desirable, when supplying polyester (A) and polyether imide (B) to a 2 axis extruding kneading machine, and carrying out melting extrusion to this pelletizing makes polyester (A) and polyether imide (B) compatibility-ize and it obtains a film of this invention.

[0042]As an extension form of a biaxially oriented polyester film of this invention, After extending to a longitudinal direction, a serial biaxial-stretching method which combined extension of every one way, such as the method of extending crosswise, a simultaneous biaxial-stretching method which extends a longitudinal direction and the cross direction simultaneously, a method which combined a biaxial-stretching method and a simultaneous biaxial-stretching method further one by one, etc. are included.

[0043]In this invention, extension temperature in a case of extending to polyester film, Although not limited,

especially when extending to an unstretched film, it is preferred to maintain at ** (glass transition temperature (Tg) of polyester film) - (Tg+120) **, and ** (Tg+10) - (Tg+80) ** are more preferred. Orientation according [ extension temperature ] to extension less than [ Tg** ] progresses too much, and it becomes difficult to extend to high magnification.

[0044]Although an example of a manufacturing method of a biaxial orientation polyester film of this invention is explained, it is not limited to this. Here, although an example using polyether imide "Ultem" as polyether imide (B) is shown using polyethylene terephthalate as polyester (A), details of manufacturing conditions change with polyester and polyether imides to be used.

[0045]First, in accordance with a conventional method, it esterifies from terephthalic acid and ethylene glycol, or bis-beta **HIDOROKISHI ethyl terephthalate (BHT) is obtained for dimethyl terephthalate and ethylene glycol by an ester exchange reaction. Next, this BHT is heated at 280 ** under a vacuum, shifting to a polymerization tank, and a polymerization reaction is advanced. Here, polyester whose intrinsic viscosity is about 0.5 is obtained. Polyether imide of the specified quantity may be added at this time. Under decompression, solid state polymerization of the obtained polyester is carried out by a pellet type. When carrying out solid state polymerization, after carrying out preliminary crystallization at temperature of 180 ** or less beforehand, solid state polymerization is carried out under decompression of about 1 mmHg at 190-250 ** for 10 to 50 hours. A method of making ethylene glycol distribute particles in a form of a slurry with specified proportion, and polymerizing this ethylene glycol with terephthalic acid as a method of making polyester which constitutes a film containing particles, is preferred. hydrosol obtained in particles at the time of composition, for example when adding particles, and alcohol -- a grain child's dispersibility added without once drying sol is good. A method of mixing water slurry of particles with a direct predetermined polyester pellet, and scouring in polyester using a vent type biaxial extruding kneading machine is also effective. A method of making a master of high-concentration particles with a described method as a method of adjusting content of particles and the number, diluting it with polyester which does not contain particles substantially at the time of film production, and adjusting content of particles is effective.

[0046]Next, it mixes at a fixed rate, and melting extrusion of a pellet (A) of this polyethylene terephthalate and the pellet (B) of polyether imide is supplied and carried out to a biaxial vent-type extruding kneading machine heated by 270-320 **. A shear rate at this time has preferred 50 to 300-$sec^{-1}$, as for 100 to 200-$sec^{-1}$, and holding time, 0.5 to 15 minutes is more preferably preferred, and they are the conditions for 1 to 10 minutes more preferably. When not dissolving on the above-mentioned conditions, extrusion may be repeated until it supplies an obtained chip to a twin screw extruder again and dissolves. By the above-mentioned kneading, it can dissolve and polyethylene terephthalate and the polyether imide can obtain a pellet of polyester with a single glass transition point.

[0047]After carrying out vacuum drying of the pellet of polyester of obtained polyether imide content at 180 ** for 3 hours or more, it is supplied to an extrusion machine heated by 280-320 ** under a nitrogen air current or a vacuum so that intrinsic viscosity may not fall, and is produced by a method currently performed from the former. In order to remove a foreign matter and deterioration polymer, it is preferred to use a filter which consists of raw materials, such as various kinds of filters, for example, a sintered metal, a porous ceramic, a sand, and a wire gauze. If needed, in order to raise constant feeding nature, a gear

pump may be formed. In the case of a laminated film, a sheet which laminated polyester and polyester of a molten state, and a mixture of polyether imide is extruded from a slit shape die using two or more sets of extrusion machines, a manifold, or a unification block, it cools on a casting roll in it, and an unstretched film is made in it.

[0048]Next, biaxial stretching of this unstretched film is carried out, and biaxial orientation is carried out. As an extension method, a biaxial-stretching method or a simultaneous biaxial-stretching method can be used one by one. Here, a serial biaxial-stretching method which performs a longitudinal direction first and then extends the cross direction is used. Although it changes with polyester (A), a structural constituent of polyether imide (B), and constituents of lamination about extension temperature, a monolayer illustrates and explains a case where it consists of polyethylene terephthalate and mixed polymer of polyether imide "Ultem" (registered trademark), for example. An unstretched film is heated by an 80-150 ** heating roller group, and it extends 1 to 10 times to a longitudinal direction in one step or two steps or more of multistage, and cools by a 20-50 ** cooling roller group. Although it is preferred to carry out in the range for /1000 to 50000% as for a longitudinal direction stretching speed, it is not limited in particular. Then, as a crosswise extension method, a method of, for example, using a tenter is common. Although it is preferred for a stretching speed to perform crosswise draw magnification by one to 10 times, to perform it by a part for /1000 to 20000%, and to perform temperature in 80-150 **, it is not limited in particular. Furthermore, re-length extension and/or re-lateral orientation are performed if needed. As extension conditions in that case, extension of a longitudinal direction is not limited in particular, although one 1.1 to 2.0 times the draw magnification [ temperature of 80-180 ** and ] of this and a method of using a tenter as a crosswise extension method are preferred and it is preferred to carry out by one 1.1 to 2.0 times the draw magnification [ temperature of 80-200 ** and ] of this. As for total draw magnification, it is preferred to a longitudinal direction that they are three to 10 times to three to 10 times and the cross direction. More preferably, it is 3.5 to 9 times to 3.5 to 9 times, and the cross direction at a longitudinal direction, and they are four to 8 times to four to 8 times, and the cross direction still more preferably at a longitudinal direction. It is preferred to extend extension temperature with slight height gradually in two or more steps in the second half of a stretching process. Then, 190-240 ** 180-250 ** is a 200-230 ** temperature requirement still more preferably preferably as a back stretching process to this oriented film, Even if there is little longitudinal direction or cross direction, it extends 1.2 to 1.5 times still more preferably 1.15 to 1.8 times preferably 1.1 to twice to one way. By giving this back stretching process, the stretch orientation of the polyether imide can fully be carried out, and the purpose of this invention can be attained. It heat-treats loosening the bottom of stress, or crosswise. Although 150 ** - 250 ** of heat treatment temperature [ 170-230 ** of ] in this case is 180-220 ** still more preferably preferably and it is preferred to carry out in the range for 0.2 to 30 seconds as for time, it is not limited in particular. Relaxation processing is preferably performed the straight side and crosswise in 1 to 6% of range to the cross direction by a cooling process from heat treatment temperature in a 100-220 ** temperature requirement. One step may be available for relaxation processing, and it may be performed in multistage and may provide change of temperature distribution. Then, if necessary [ to a room temperature ] in a film, performing relaxation processing the straight side and crosswise, a film will be cooled and rolled round and the target biaxial orientation polyester film will be obtained.

[0049][A measuring method of physical properties and valuation method of an effect] A measuring method

of weighted solidity and the valuation method of an effect are as follows.

[0050](1) The measuring condition of laser Raman strength by laser Raman strength laser Raman scattering process by laser Raman scattering process is as follows.

Laser Raman device Ramanor T-64000 (made by Jobin Yvon)

Microprobe Object lens x100. crossing slit : 400 micrometers spot diameter: -- 1 micrometer. Light source Ar+ laser :. [ NEC GLG3460 ] 5145A Output : 100 mW Spectroscope Elements of the Invention :. [ 640 mm ] Triple Monochromator diffraction grating :P AC. Holographic 76x76mm Premonochromator 1800g/mm Spectrograph 1800g/mm distribution: Single 7A/mm detector CCD (Jobin Yvon 1024x256)

A film used for measurement was sampled and took out a section with a microtome to an epoxy resin after embedding. A section adjusted a thing parallel to a longitudinal direction, the cross direction or a longitudinal direction, the cross direction, and a direction that makes 45 degrees, and measurement shifted a position, measured it 10 times and took average value. a ratio of Raman peak intensity (I) of 1776-$cm^{-1}$ in polarization with measurement parallel to a film plane to the Raman peak intensity (IND) of 1776-$cm^{-1}$ in polarization vertical to a film plane -- I/IND was calculated and it was considered as an index of orientation of polyether imide. Similarly, from the Raman peak intensity of 1615-$cm^{-1}$, it asked for an intensity ratio and was considered as an index of orientation of polyester.

[0051](2) On condition of the following, it measured by the diffractometer method using half-peak-width X-ray diffractometer (Rigaku Corp. Make 4036A2 type (bulb type)) of a circumferencial direction of a crystal face diffraction peak of a film by a wide angle X-ray diffraction method.

X-ray diffractometer Rigaku Corp. Make 4036A2 type (bulb type)

X line source : CuK alpha rays (nickel filter use)

output :40kV 20mA goniometer Rigaku Corp. Make Slit : 2mmphi-1 degree-1 degree detector : Scintillation counter calculation -- recorder Rigaku Corp. Make In a diffraction peak position of a crystal face acquired with a RAD-C type 2 theta/theta scan. It starts to 2 cm x 2 cm, a sample and a counter on top of which a direction was arranged and laid are fixed, and a profile of a circumferencial direction is obtained by carrying out the field internal version of the sample (beta scan). The full width at half maximum (deg) was calculated by making a valley part of both ends of a peak into a background among peak profiles obtained with beta scan.

[0052](3) On condition of the following, it measured with a penetration method using crystal size X-ray diffractometer (Rigaku Corp. Make 4036A2 type) obtained from a wide angle X-ray diffraction method.

X-ray diffractometer Rigaku Corp. Make 4036A2 type X line source : CuK alpha rays (nickel filter use)

output :40kV 20mA goniometer Rigaku Corp. Make Slit : 2mmphi-1 degree-1 degree detector : Scintillation counter calculation -- recorder Rigaku Corp. Make RAD-C type.

[0053]It started to 2 cm x 2 cm, a direction was arranged and piled up, a sample hardened with a collodion ethanol solution was set, and it calculated from half breadth of an all directions-oriented field using a formula of following Scherrer among 2 theta/theta strength data obtained by wide angle X diffraction measurement. A crystal size measured an orientation main axis direction here.

Crystal size L(angstrom) =Klambda/$beta_0$cos$theta_B$K : Constant (=1.0)

lambda : -- wavelength (= 1.5418 A) of X-rays

$theta_B$: Bragg-angle $beta_0=(beta_E^2-beta_I^2)^{1/2}$ $beta_E$: Apparent half breadth (actual measurement)

$beta_I$: Device constant (= $1.046x10^{-2}$).

[0054](4) extrapolation glass transition starting temperature (Tg-onset) and glass transition temperature (Tg)
Specific heat measurement was performed on the following device and conditions by a false isothermal method, and it determined according to JIS K7121.
device : -- product temperature abnormal-conditions DSC measurement condition [ made by TA Instrument ]: -- cooking temperature : 270-570K (RCS cooling method)
temperature proofreading : melting point temperature modulation amplitude [ of high grade indium and tin ]: -- **1K temperature modulation period: -- 60-second temperature-up step: -- 5K sample weight : 5-mg specimen container : Open sand mold container (22 mg) made from aluminum
Reference container : Open sand mold container made from aluminum (18 mg)
Glass transition temperature is a following formula. Glass transition temperature = (extrapolation glass transition starting temperature + extrapolation glass transition finish temperature) it computed by /2.
[0055](5) In accordance with a method specified to Young's modulus ASTM-D882, it measured using an Instron type tension tester. Measurement was made into the following conditions.
measuring device: -- a product film [ made from Cage Ene Tech ] strong ductility automatic measuring instrument "tensilon AMF/RTA-100" -- specimen size: -- 100 mm, a tension speed:200-mm part temperature [ measurement environment:] of 23 ** for /, and 65% of humidity RH between 10 mm[ in width ] x trial length.
[0056](6) It measured according to heat shrinkage rate JIS-C2318.
Specimen size: 10 mm in width, and temperature of 100 **, processing time 30 minutes and 100 ** of 200 mm of marked-line interval measuring condition:no load condition heat shrinkage rate were searched for from a following formula.
The marked-line interval L before heat-shrinkage-rate (%) $=[(L_0-L)/L_0]$ $x100L_0$:heat-treatment: A marked-line interval after heat-treatment.
[0057](7) Use a value calculated from a lower type among intrinsic viscosity alt.chlorophenol from solution viscosity measured at 25 **. namely, etasp/C=[eta]+K[eta]2 and C -- here, dissolution polymer weight per 100 ml of solvents (g/100ml, usually 1.2) and K of etasp=(solution viscosity / solvent viscosity)-1 and C are Huggins constants (referred to as 0.343). Solution viscosity and solvent viscosity were measured using an Ostwald viscometer.
[0058](8) The magnetic parametric performance (C/N) of magnetic tape
Multistory spreading (the upper layer changed suitably thickness of 0.1 micrometer of coating thickness and a nonmagnetic lower layer with a magnetic paint) is carried out by an extrusion coater, and the surface of polyester film of this invention is made to carry out magnetic orientation of a magnetic paint and a nonmagnetic coating material of the following presentation, and is made to dry them. Subsequently, after forming a back coat layer of the following presentation in an opposite side and carrying out a calendar process by temperature:85 ** and linear pressure:200kg/cm with a small test calendar device (steel/steel roller, five steps), curing is carried out at 60 ** for 48 hours. The slit of the above-mentioned tape original

fabric was carried out to 8-mm width, and a pancake was created. Subsequently, length 200 m minutes were included in a cassette from this pancake, and it was considered as a cassette tape.
[0059]Commercial VTR for Hi8 ( EV-BS3000 by SONY) was used for this tape, and 7MHz+1MHz C/N (a career versus noise ratio) was measured. As compared with videotape for Hi8 of marketing of this C/N (120-minute MP by SONY), judged not less than +3 dB to be O, +1 or more dB [ less than +3 ] was judged to be **, and less than +1 dB was judged to be x. O Although it is desirable, it is usable practical also in **.
(Presentation of a magnetic paint)
- ferromagnetic metal powder : 100 weight sections - sulfonic acid Na conversion vinyl chloride copolymer : Ten weight sections - sulfonic acid Na conversion polyurethane : Ten weight sections - polyisocyanate : Five weight sections - stearic acid : 1.5 weight sections . - Oleic acid : One weight section - carbon black : One weight section - alumina : Ten weight sections - methyl ethyl ketone : 75 weight sections - cyclohexanone : 75 weight sections - toluene : 75 weight sections (presentation of a nonmagnetic lower layer paint)
- titanium oxide : 100 weight sections - carbon black . : Ten weight sections - sulfonic acid Na conversion vinyl chloride copolymer : Ten weight sections - sulfonic acid Na conversion polyurethane : Ten weight sections - methyl ethyl ketone : 30 weight sections - methyl isobutyl ketone : 30 weight sections and toluene : 30 weight sections (presentation of a back coat)
- carbon black (mean particle diameter of 20 nm). : 95 weight sections - carbon black (mean particle diameter of 280 nm): 10 weight section - alpha alumina : 0.1 weight section - zinc oxide : 0.3 weight section - sulfonic acid Na conversion polyurethane : 20 weight sections - sulfonic acid Na conversion vinyl chloride copolymer . : 30 weight sections - cyclohexanone : 200 weight sections - methyl ethyl ketone : 300 weight sections - toluene : 100 weight sections.
[0060](9) Apply, make the surface of the running durability of magnetic tape, and a biaxial orientation polyester film of preservability this invention carry out magnetic orientation of the magnetic paint of the following presentation so that it may be set to spreading 2.0 micrometers in thickness, and make it dry it. Subsequently, after forming a back coat layer of the following presentation in an opposite side and carrying out a calendar process, curing is carried out at 70 ** for 48 hours. The slit of the above-mentioned tape original fabric was carried out to 1/2-inch width, and as magnetic tape, length 670 m minutes were included in a cassette, and was used as a cassette tape.
(Presentation of a magnetic paint)
- ferromagnetic metal powder : 100 weight sections - conversion vinyl chloride copolymer : Ten weight sections - conversion polyurethane : Ten weight sections - polyisocyanate : Five weight sections - stearic acid : 1.5 weight sections - oleic acid : . One weight section - carbon black : One weight section - alumina : Ten weight sections - methyl ethyl ketone : 75 weight sections - cyclohexanone : 75 weight sections - toluene : 75 weight sections (presentation of a back coat)
- carbon black (mean particle diameter of 20 nm). : 95 weight sections - carbon black (mean particle diameter of 280 nm): 10 weight section - alpha alumina : 0.1 weight section - conversion polyurethane : 20 weight sections - conversion vinyl chloride copolymer : 30 weight sections - cyclohexanone . : 200 weight sections and methyl ethyl ketone : 300 weight sections - toluene : A cassette tape which carried out 100 weight-section creation, Using product Magstarmade by IBM 3590 MODELB1A Tape Drive, the both-way run was carried out 100 times, and the following standard estimated the running durability of a tape. O It

was considered as an acceptable product.
O : there is nothing, it can delete elongation of a tape end side, and bending, and marks are not seen.
**: Although there are not elongation of a tape end side and bending, a part can be deleted and marks are seen.
x: A part of tape end side is extended, modification of the shape of wakame seaweed can be seen and deleted, and marks are seen.
[0061]A cassette tape which created [ above-mentioned ] to product Magstarmade by IBM 3590 MODELB1A Tape Drive. After reading data and saving a cassette tape in atmosphere of 40 ** and 80%RH for 100 hours, data was played and the following standard estimated the preservability of a tape. O It was considered as an acceptable product.
O : there is also no track gap and it reproduced normally.
**: Although there are no abnormalities in tape width, it reads in part and a failure is seen.
x: Tape width has change and a reading failure is seen.
[0062](10) Coating of the hot printing ink of the following presentation in polyester film for thermal transfer ribbons of this invention which applied a weld prevention layer to printing nature one side of a thermal transfer ribbon was carried out to a weld prevention layer by a hot melt coating machine in an opposite side so that coating thickness might be set to 3.5 micrometers, and a thermal transfer ribbon was created.
(Presentation of hot printing ink)
Carnauba wax : 60.6 % of the weight Microcrystallin wax : 18.2-% of the weight vinyl acetate ethylenic copolymer : 0.1 % of the weight Carbon black : About a thermal transfer ribbon created 21.1% of the weight. Black solid was printed with a bar code printer made from Oaks (BC-8), and printing nature was evaluated. O It was considered as an acceptable product.
O : print vividly.
**: A pitch gap arises in printing.
x: Wrinkles go into a ribbon and printing is confused.
xx: Wrinkles go into a film at the time of hot melt coating, and hot printing ink cannot apply uniformly.
[0063](11) Characterization insulation resistance for capacitors and dielectric breakdown voltage were evaluated as follows.
[0064]A. Vacuum deposition of the aluminum was carried out so that a surface resistance value might become 2ohms / ** at one side of polyester film of insulation resistance this invention. It vapor-deposited to stripe shape which has a margin part which runs to a longitudinal direction at that time (repetition 57 mm in width of a deposition part, and 3 mm [ of a margin part ] in width). Next, the slit of the edge was put in and carried out in a center of each deposition part, and the center of each margin part, and it was considered as a take up reel of 30 mm [ in overall width ] tape shape which has a margin of 1.5-mm width on the left or the right. One pair of aluminum deposition film which has the obtained symmetrical margin was piled up, and it wound around length used as capacity of 1.5 micro F. This winding article was pressed for 10 minutes, and was fabricated by a pressure of 120 ** and 20 $kg/cm^2$. Thermal spraying of Metallikon was carried out, it was considered as an electrode, a lead was attached to a both-ends side, and it was considered as a capacitor sample. Here 1000 created 1.5-micro F capacitor samples Subsequently, 23 **, Under atmosphere of 65%RH, it measured as a 1-minute value in the impressed electromotive force 500V

with the superinsulation ohm-meter 4329A made from YHP, insulation resistance used a capacitor sample below 5000 M omega as inferior goods, and it judged on the following standards. In this invention, O, O, and ** were considered as success.

O : -- inferior goods -- less than [ ten piece ] O: -- inferior goods -- less than [ ten or more pieces / 20 ] **: -- inferior goods -- less than [ 20 or more pieces / 50 ] x: -- inferior goods -- 50 or more pieces.

[0065]B. Evaluate as follows, using as a specimen a film which has not performed metal deposition to dielectric-breakdown-voltage JIS-C-2318 according to a method of a statement.

[0066]It covers with one rubber plate (rubber Shore hardness of about 60 degrees, and about 2 mm in thickness) on metal plates of a suitable size, What piled up ten aluminium foil about 6 micrometers thick on it is used as a lower electrode, it is smooth and the bottom with a diameter of 8 mm which had a radius of circle of about 1 mm on the outskirts in a weight of about 50 g uses a pillar made from brass without a crack as an upper electrode. A specimen is beforehand neglected for 48 hours or more in atmosphere of temperature of 20**5 **, and 65**5% of relative humidity. A specimen is put between an upper electrode and a lower electrode, and direct current voltage is impressed by DC power supply between two electrodes in atmosphere of temperature of 20**5 **, and 65**5% of relative humidity, and it is made to go up until it carries out the dielectric breakdown of this direct current voltage from 0V with speed of 100V in 1 second. It examines to 50 samples, average value of what **(ed) dielectric breakdown voltage by thickness of a specimen is calculated, and the value considers micrometer as success (O) in not less than 400v /.

[0067]

[Example]The embodiment of this invention is described based on the following example.

[0068]Example 1 -- the pellet of the polyethylene terephthalate (intrinsic viscosity 0.85) obtained by the publicly known method -- the pellet of 50 % of the weight and polyether imide -- " -- 1010"(GE plastics company registered trademark) 50 % of the weight of Ultem, The biaxial vent-type extruding kneading machine heated by 300 ** was supplied, and polyester chip (I) which contained melt extruding and polyether imide 50% of the weight in 100 sec of shear rate $^{-1}$, and holding time 1 minute was obtained. it mixing with 60 % of the weight of pellets of polyethylene terephthalate (intrinsic viscosity -- zero . -- 62 -- a sliding agent -- ****** -- with the spherical bridge construction polystyrene particle with a pitch diameter of 0.3 micrometer of 0.2 % of the weight, and a pitch diameter of 0.8 micrometer 0.01 % of the weight of spherical bridge construction polystyrene particle combination), and this chip (I)40 % of the weight, The biaxial vent-type extruding kneading machine heated by 280 ** was supplied, and polyester chip (II) which contained melt extruding and polyether imide 20% of the weight in 100 sec of shear rate $^{-1}$, and holding time 1 minute was obtained. The obtained chip is transparent and only a single glass transition temperature was observed.

[0069]On the other hand, 40 % of the weight and the pellet of polyethylene terephthalate (with the intrinsic viscosity 0.62 and a pitch diameter of 0.07 micrometer 0.16 % of the weight of spherical-silica-particles combination) for polyester chip (I) 60 % of the weight, The biaxial vent-type extruding kneading machine heated by 280 ** was supplied, and polyester chip (III) was obtained in the similar way. The obtained chip is transparent and only a single glass transition temperature was observed.

[0070]To the extrusion machine A heated by 280 **, using two extrusion machines. To the extrusion machine B which supplied after carrying out vacuum drying of the pellet of obtained polyether imide

content polyester composition (III) at 180 ** for 3 hours, and was similarly heated by 280 **. It supplies, after carrying out vacuum drying of the pellet of obtained polyether imide content polyester composition (II) at 180 ** for 3 hours, Adhesion cooling solidification was carried out making it join in a T die (lamination ratio II/III/II=1/10/1), and making electrostatic charge impress to a cast drum with a skin temperature of 25 ** in order to laminate three layers so that polyester composition (II) may become the outermost layer, and the lamination unstretched film was created.

[0071]With the roll type drawing machine, in one step, this unstretched film was extended 3.2 times at the temperature of 105 ** to the longitudinal direction, and was further extended crosswise 3.8 times at the temperature of 95 ** using the tenter to it. Then, with the roll type drawing machine, it extended to the longitudinal direction in two steps, re-extended 1.5 times at the temperature of 150 **, and re-extended 1.2 times at the temperature of 200 ** crosswise using the tenter. It extended 1.2 times at the temperature of 220 ** crosswise. 1% of relaxation processing was performed crosswise after heat treatment for 10 seconds at the temperature of 220 ** under fixed length, and the 5-micrometer-thick laminated polyester film was obtained.

[0072]A presentation, the characteristic, etc. of this biaxial orientation polyester film are as having been shown in Table 1 and 2, and had the characteristic outstanding as a film of the various applications for magnetic recording media etc.

[0073]After having changed the content of polyether imide like example 2 Example 1 as shown in Table 1, and obtaining a polyether imide content polyester composition, the biaxial orientation polyester film was obtained by the same method as Example 1.

[0074]The characteristic of this biaxial orientation polyester film had the characteristic outstanding as a film of the various applications for magnetic recording media etc. as it was shown in Table 2.

[0075]the unstretched film after obtaining an unstretched film like example 3 Example 1 -- with the roll type drawing machine, extension and the temperature of 90 ** performed extension to the longitudinal direction 2.5 times twice at the temperature of 120 ** in two steps, and it extended 3.3 times at the temperature of 95 ** crosswise further using the tenter. Then, with the roll type drawing machine, it extended to the longitudinal direction in two steps, re-extended 1.3 times at the temperature of 155 **, and re-extended 1.2 times at the temperature of 190 ** crosswise using the tenter. It extended 1.2 times at the temperature of 220 ** crosswise. 1% of relaxation processing was performed crosswise after heat treatment for 10 seconds at the temperature of 220 ** under fixed length, and the 5-micrometer-thick laminated polyester film was obtained.

[0076]The characteristic of this biaxial orientation polyester film had the characteristic outstanding as a film of the various applications for magnetic recording media etc. as it was shown in Table 2.

[0077]After obtaining an unstretched film like example 4 Example 1, the unstretched film was performed to the longitudinal direction in one step, the roll type drawing machine performed extension twice at the temperature of 120 **, and it extended twice at extension and the temperature of 90 ** twice with the temperature of 120 ** crosswise further using the tenter. Then, with the roll type drawing machine, it extended to the longitudinal direction in two steps, re-extended 3.7 times at the temperature of 90 **, and the temperature of 135 **, and re-extended 1.2 times at the temperature of 190 ** crosswise using the tenter. It extended 1.2 times at the temperature of 220 ** crosswise. 1% of relaxation processing was performed crosswise after heat treatment for 10 seconds at the temperature of 220 ** under fixed length,

and the 5-micrometer-thick laminated polyester film was obtained.

[0078]The characteristic of this biaxial orientation polyester film had the characteristic outstanding as a film of the various applications for magnetic recording media etc. as it was shown in Table 2.

[0079]Example 5 -- the polyethylene 2 obtained by the publicly known method, and 6-naphthalate (PEN) (the intrinsic viscosity 0.65 and the glass transition temperature of 125 **) The pellet of with the spherical bridge construction polystyrene particle with a pitch diameter of 0.3 micrometer of 0.2 % of the weight, and a pitch diameter of 0.8 micrometer 0.01 % of the weight of spherical bridge construction polystyrene particle combination 1010"(GE plastics company registered trademark) 20 % of the weight of pellet" Ultem of 80 % of the weight and polyether imide, The biaxial vent-type extruding kneading machine heated by 290 ** was supplied, and polyester chip (IV) which contained melt extruding and polyether imide 20% of the weight in 100 sec of shear rate $^{-1}$, and holding time 1 minute was obtained. The obtained chip is transparent and only a single glass transition temperature was observed.

[0080]On the other hand, they are the polyethylene 2 and 6-naphthalate (PEN) (the intrinsic viscosity 0.65 and the glass transition temperature of 125 **). The pellet of 0.16 % of the weight of spherical-silica-particles combination with a pitch diameter of 0.07 micrometer 1010"(GE plastics company registered trademark) 20 % of the weight of pellet" Ultem of 80 % of the weight and polyether imide, The biaxial vent-type extruding kneading machine heated by 300 ** was supplied, and polyester chip (V) which contained polyether imide 20% of the weight in the similar way was obtained. The obtained chip is transparent and only a single glass transition temperature was observed.

[0081]To the extrusion machine A heated by 290 **, using two extrusion machines. To the extrusion machine B which supplied after carrying out vacuum drying of the pellet of obtained polyether imide content polyester composition (V) at 180 ** for 3 hours, and was similarly heated by 290 **. It supplies, after carrying out vacuum drying of the pellet of obtained polyether imide content polyester composition (IV) at 180 ** for 3 hours, Adhesion cooling solidification was carried out making it join in a T die (lamination ratio IV/V/IV=1/10/1), and making electrostatic charge impress to a cast drum with a skin temperature of 25 ** in order to laminate three layers so that polyester composition (IV) may become the outermost layer, and the lamination unstretched film was created.

[0082]With the roll type drawing machine, in one step, this unstretched film was extended 5.0 times at the temperature of 145 ** to the longitudinal direction, and was further extended crosswise 5.0 times at the temperature of 150 ** using the tenter to it. It extended 1.1 times at the temperature of 220 ** crosswise. 1% of relaxation processing was performed crosswise after heat treatment for 10 seconds at the temperature of 220 ** under fixed length, and the 5-micrometer-thick laminated polyester film was obtained.

[0083]A presentation, the characteristic, etc. of this biaxial orientation polyester film are as having been shown in Table 1 and 2, and had the characteristic outstanding as a film of the various applications for magnetic recording media etc.

[0084]In 13 layers of comparative example lamination (II/III/II), the unstretched film was created like Example 1 except making it the polyethylene terephthalate (PET) with which three layers of polyether imides are not mixed.

[0085]With the roll type drawing machine, in one step, this unstretched film was extended 3.2 times at the

temperature of 95 ** to the longitudinal direction, and was further extended crosswise 3.8 times at the temperature of 95 ** using the tenter to it. Then, with the roll type drawing machine, it extended to the longitudinal direction in two steps, re-extended 1.5 times at the temperature of 135 **, and re-extended 1.2 times at the temperature of 190 ** crosswise using the tenter. It extended 1.2 times at the temperature of 220 ** crosswise. 1% of relaxation processing was performed crosswise after heat treatment for 10 seconds at the temperature of 220 ** under fixed length, and the 5-micrometer-thick laminated polyester film was obtained.

[0086]This biaxial orientation polyester film does not contain polyimide ether, and its presentation, characteristic, etc. are inferior as a film of the various applications for magnetic recording media etc. as they were shown in Table 1 and 2.

[0087]After having changed the content of polyether imide like comparative example 2 Example 1 as shown in Table 1, and obtaining a polyether imide content polyester composition, the biaxial orientation polyester film was obtained by the same method as Example 1.

[0088]The Raman peak intensity ratio measured with laser Raman scattering process is outside the range of this invention as this polyester film was shown in Table 1.

The characteristic is inferior as a film of the various applications for magnetic recording media etc. as it was shown in Table 2.

[0089]The 5-micrometer-thick laminated polyester film was obtained like Example 1 except not giving 1.2 time extension at the temperature of 220 ** of the comparative example 3 last.

[0090]The Raman peak intensity ratio measured with laser Raman scattering process is outside the range of this invention as this polyester film was shown in Table 1.

The characteristic is inferior as a film of the various applications for magnetic recording media etc. as it was shown in Table 2.

[0091]50% of the weight of polyether imide content polyester chip (I) obtained like example 6 Example 1 40 % of the weight, Mix with 60 % of the weight of pellets of polyethylene terephthalate (with the intrinsic viscosity 0.62 and a pitch diameter of 1.0 micrometer 0.2 % of the weight of silica dioxide particle combination), and the biaxial vent-type extruding kneading machine heated by 290 ** is supplied, Polyester chip (X) which contained melt extruding and polyether imide 20% of the weight in 100 sec of shear rate $^{-1}$, and holding time 1 minute was obtained. The obtained chip is transparent and only a single glass transition temperature was observed.

[0092]After carrying out vacuum drying of this polyester chip (X) at 180 ** for 3 hours, the extrusion machine heated by 280 ** was supplied and it breathed out from melt extruding and a T die to the sheet shaped. Furthermore, this sheet was stuck to cooling drum lifting with a skin temperature of 25 ** by electrostatic force, cooling solidification was carried out, and the unstretched film was obtained. Coating was carried out by the photogravure coating machine so that the coating thickness after drying the ointment of the following presentation as a weld prevention layer on one side of this unstretched film might be set to 0.5 micrometer.

(Presentation of an ointment)

Acrylic ester : 14.0 % of the weight Amino modifying silicone : 5.9 % of the weight Isocyanate : 0.1 % of the weight Water : 80.0 % of the weight.

[0093]Then, it extended on the same extension conditions as Example 1 to the obtained unstretched film, processing for thermal transfer ribbons was performed to the film with a thickness of 4 micrometers obtained further, and the practical use characteristic as an object for thermal transfer ribbons was evaluated. The result had the outstanding characteristic as Table 3.

[0094]The unstretched film was obtained like Example 6 except using the polyethylene terephthalate which does not contain comparative example 4 polyether imide. This unstretched film was extended like the comparative example 1, and the biaxial orientation polyester film was obtained. Processing for thermal transfer ribbons was performed to the film with a thickness of 4 micrometers obtained, and the practical use characteristic as an object for thermal transfer ribbons was evaluated. A result is inferior to the practical use characteristic as Table 3.

[0095]50% of the weight of polyether imide content polyester chip (I) obtained like example 7 Example 1 40 % of the weight, Mix with 60 % of the weight of pellets of polyethylene terephthalate (with the intrinsic viscosity 0.62 and a pitch diameter of 1.2 micrometers 0.1 % of the weight of condensation silica particle combination), and the biaxial vent-type extruding kneading machine heated by 290 ** is supplied, Polyester chip (XI) which contained melt extruding and polyether imide 20% of the weight in 100 sec of shear rate $^{-1}$, and holding time 1 minute was obtained. The obtained chip is transparent and only a single glass transition temperature was observed.

[0096]After carrying out vacuum drying of this polyester chip (XI) at 180 ** for 3 hours, the extrusion machine heated by 280 ** was supplied and it breathed out from melt extruding and a T die to the sheet shaped. Furthermore, this sheet was stuck to cooling drum lifting with a skin temperature of 25 ** by electrostatic force, cooling solidification was carried out, and the unstretched film was obtained. To this unstretched film, the film with a thickness of 4 micrometers which extended on the same extension conditions as Example 1 was processed into capacitors, and the practical use characteristic was evaluated. The result had the outstanding characteristic as Table 4.

[0097]The unstretched film was obtained like Example 7 except using the polyethylene terephthalate which does not contain comparative example 5 polyether imide. This unstretched film was extended like the comparative example 1, and the biaxial orientation polyester film was obtained. The film with a thickness of 4 micrometers obtained was processed into capacitors, and the practical use characteristic was evaluated. A result is inferior to the practical use characteristic as Table 4.

[Table 1]

| | ラマンピーク強度比 1776cm$^{-1}$ ($I_{MD}/I_{ND}$) / ($I_{TD}/I_{ND}$) | ラマンピーク強度比 1776cm$^{-1}$ ($I_{MD}/I_{46}$) / ($I_{TD}/I_{46}$) | ラマンピーク強度比 1615cm$^{-1}$ ($I_{2MD}/I_{2ND}$) / ($I_{2TD}/I_{2ND}$) | 結晶配向解析による半値幅 (度) | 結晶サイズ (オングストローム) | ポリエステル(A)とポリエーテルイミド(B)からなる成分 Tg onset (℃) | ポリエーテルイミド量 (重量%) |
|---|---|---|---|---|---|---|---|
| 実施例1 | 4.0/3.5 | 2.7/2.2 | 9.5/9.8 | 38 | 55 | 102 | 20 |
| 実施例2 | 3.2/3.0 | 2.5/1.9 | 9.0/9.2 | 37 | 50 | 95 | 5 |
| 実施例3 | 5.2/3.7 | 3.2/2.0 | 11.5/8.8 | 44 | 57 | 102 | 20 |
| 実施例4 | 3.8/5.4 | 2.1/3.4 | 9.0/12.3 | 76 | 65 | 102 | 20 |
| 実施例5 | 4.2/3.7 | 2.8/2.3 | - | 35 | 41 | 140 | 20 |
| 実施例6 | 3.7/3.5 | 2.5/2.1 | 9.2/9.2 | 40 | 54 | 102 | 20 |
| 実施例7 | 3.6/3.5 | 2.4/2.1 | 9.3/9.1 | 41 | 54 | 102 | 20 |
| 比較例1 | - | - | 9.2/9.3 | 37 | 50 | 80 | 0 |
| 比較例2 | 1.4/1.3 | 1.3/1.2 | 7.2/6.8 | 45 | 46 | 117 | 40 |
| 比較例3 | 1.9/1.8 | 1.4/1.3 | 4.8/4.7 | 74 | 48 | 102 | 20 |
| 比較例4 | - | - | 9.2/9.3 | 38 | 49 | 80 | 0 |
| 比較例5 | - | - | 9.3/9.3 | 38 | 50 | 80 | 0 |

(注) MD/TD：フィルム長手方向/幅方向

[Table 2]

| | ヤング率 MD/TD(和) [GPa] | 熱収縮率 (100℃, 30分) MD/TD [%] | 電磁変換特性 | 走行耐久性 | 保存性 |
|---|---|---|---|---|---|
| 実施例1 | 6.7/6.5(13.2) | 0.3/0.2 | ○ | ○ | ○ |
| 実施例2 | 6.6/6.5(13.1) | 1.0/0.8 | ○ | ○ | ○ |
| 実施例3 | 7.2/4.3(11.5) | 0.3/0.1 | ○ | ○ | ○ |
| 実施例4 | 6.3/7.8(14.1) | 0.1/0.2 | ○ | ○ | ○ |
| 実施例5 | 7.2/8.6(15.8) | 0.1/0.1 | ○ | ○ | ○ |
| 比較例1 | 6.5/6.3(12.8) | 2.5/1.8 | △ | △ | × |
| 比較例2 | 5.7/5.8(11.5) | 0.2/0.2 | × | × | ○ |
| 比較例3 | 5.5/5.9(11.4) | 0.3/0.2 | × | × | ○ |

(注) MD/TD：フィルム長手方向/幅方向

[Table 3]

| | ヤング率 MD/TD(和) [GPa] | 熱収縮率 (100℃, 30分) MD/TD [%] | 印字性 |
|---|---|---|---|
| 実施例6 | 6.5/6.7(13.2) | 0.3/0.2 | ○ |
| 比較例4 | 6.3/6.2(12.5) | 2.3/1.7 | × |

(注) MD/TD：フィルム長手方向/幅方向

[Table 4]

| | ヤング率 MD/TD(和) [GPa] | 熱収縮率 (100℃, 30分) MD/TD [%] | 絶縁抵抗 | 絶縁破壊電圧 |
|---|---|---|---|---|
| 実施例7 | 6.6/6.5(13.1) | 0.3/0.2 | ◎ | ○ |
| 比較例5 | 6.4/6.2(12.6) | 2.2/1.8 | × | × |

(注) MD/TD：フィルム長手方向/幅方向

[Effect of the Invention]According to this invention, the biaxial orientation polyester film which raised mechanical characteristics and dimensional stability, such as Young's modulus of a film, can be obtained. It is widely utilizable for the various film applications the object for magnetic recording media, the object for capacitors, for thermal-ink-transfer-printing ribbons, etc. As an object for magnetic recording media, it excels in a magnetic parametric performance, running durability, preservation stability, etc., and,

specifically, the base film which was excellent in printing nature as an object for thermal-ink-transfer-printing ribbons can be obtained further.

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[Translation done.]